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(19)

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(54) SOFTENING COMPOSITIONS

(71) We, UNILEVER LIMITED, a company organised under the laws of Great Britain, of Unilever House, Blackfriars, London E/C 4, England, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention concerns a fabric-softening composition. More particularly the present invention relates to a composition for reducing the rate of which cotton-containing fabrics become harsh or reducing the harshness of such fabrics, said composition comprising cellulolytic enzymes.

It is well known in the art that washing of fabrics can cause severe harshening of the fabrics. The degree of harshening is dependent upon a number of factors and conditions during washing operations, such as the detergent compositions used, the time and temperature of the washing operation, type of washing machine, the cloth/liquor ratio and the number of washes. This problem is particularly acute with fabrics made from cotton fibres, such as towels and napkins. Harshening of fabrics of this type has been recognized in the art, and several proposals have been made to reduce such harshening. They include treatment of the fibres after washing with a softening composition, which contains a cationic detergent as the effective agent. Such compositions also often referred to as rinse conditioners since they are normally applied in the rinsing stage of the washing operation, can have a satisfactory effect. A disadvantage of this method of alleviation of harshness of fabrics is, however, that it is only temporary, that is until the next wash, where the softening effect of the rinse conditioner is reduced.

It has been found that treatment with cellulolytic enzymes containing C_1 and C_x cellulases significantly reduces the rate at which new cotton articles become harsh and can partially restore the original softness to already harsh cotton articles. Cotton fabrics thus treated feel markedly softer after several washes than those treated with a conventional rinse conditioner. Surprisingly, furthermore. no significant fabric damage occurs under user's conditions of the compositions of the invention, although one would expect the cellulolytic enzymes to reduce the strength of the cotton fibres.

The present invention therefore provides a composition for reducing the harshness of cotton-containing fabrics, said composition comprising a cellulolytic enzyme as herein defined, and a detergent active material. Cellulolytic enzymes are well known; they are produced by fungi, for example by a submerged culture of a strain of Trichoderma on wheat bran. Other examples are fungal cellulolytic enzymes produced by cultures of Myrothecium verrucaria, Aspergillus orycae, Aspergillus niger, Botrytis cinerea. Bacterial cellulolytic enzymes are also known, e.g. produced by strains of Streptomycetes and Hymenomycetes. The cellulolytic enzymes to be used in the present invention contain two types of constituents, called Cellulase C_1 and Cellulase C_x, in which C_x is capable of splitting the β-glucosidic 1,4 bond of cellulose whereas the cellulase C_1 is not capable of doing so, C_1-cellulase is required for initial breakdown of cellulose. For further details, reference is hereby made to "Cellulases and their application", Proceedings of a symposium. American Society Special Publication 95 (1969) page 23. In this reference C_1-cellulase is defined as an enzyme whose action is unspecified. It is required for the hydrolysis of highly oriented solid cellulose by β-1,4 glucanases. The cellulolytic enzymes of the present invention may furthermore, in addition to C_1 and C_x cellulase contain minor amounts of other enzymes, such as cellobiase, xylanase, amylase, glucanase, protopectinase, maltase, saccharose, protease, lipase, pertinase and phospholipase.

Commercial C_1 and C_x cellulase-containing cellulolytic enzymes normally contain minor amounts of such other enzymes.

Cellulolytic enzymes are commercially avail-

able, e.g. "Meicelase" ex Meiji Seika Kaisha Ltd., 2 Chome Kyobashi, Chuo-ku, Tokyo, Japan, which is a cellulolytic brown enzyme powder produced by culturing Trichoderma on a solid medium of wheat or a liquid medium containing bran. The water-extracted solution of the solid culture or the filtrate of the submerged culture is processed and dried to produce a brown powder.

Other commercially available cellulolytic enzymes are Enzyme 19 AF ex Rohm and Haas Cy. a cellulolytic enzyme produced by Aspergillus niger, Cellulase ex Koch Light code 36845, cellulase C—7377 ex Sigma Corp., and cellulolytic enzymes P 500 and P 1500 ex Kinki Yakult Co. Ltd. Onozuka, Japan, made from Tichoderma viride.

It has been found that, at the same C_x content, the more C_1 present the more significant are the softening effects which can be obtained. It is believed, although this theory should not in any way be considered to be a limitation of the present invention, that C_x and C_1 coact in some way. Therefore, it is essential that the cellulolytic enzyme preparations which are used in the present invention contain both constitutes C_x and C_1.

The C_1 and C_x activities can be determined by the following method using two commercial preparations as examples. Meicelase P and Cellulase 1000 u/g ex Aspergillus niger from Koch Light Co. Ltd., code 36845 have been determined for "C_1" and for "C_x" in the following ways:

C_1 A 500 ml Erlenmeyer flask containing 10 ml of an aqueous solution of 1% of the enzyme was brought to 40°C in a shaking apparatus. The solution was buffered to pH 4 by 0.05M acetic acid/sodium acetate. Four pieces of filter paper (1cm × 1 cm; Schleicher und Schutt No. 595½) were added to the mixture. The time needed for complete disintegration of the filter paper was taken as a measure of C_1 activity.

Results:

Meicelase P	1½ hours
Cellulase ex Koch Light	not disintegrated even after 100 hours.

The quicker the filter paper is degraded, the more suitable the cellulolytic enzyme is for use in the present invention.

C_x 20 ml of a 1% sodium carboxymethylcellulose (Tylose®, ex Farbwerke Hoescht, coded C 300 P) aqueous solution buffered to pH 5 with 0.1M sodium acetate and acetic acid, was incubated for 15 minutes at 40°C with different quantities of enzyme added in 2 ml water. After the incubation the reaction was stopped by adding 1 ml of 2N NaOH solution. The viscosity of the SCMC solution was measured by means of an Ubbelohde viscometer, coded 265657, and the viscosity was expressed in seconds.

Results:	Time	Meicelase P concentration (M) % by weight	Koch Light (KL) Cellulase % by weight	Ratio KL/M
	570 secs.	0	0	
	400 secs.	$7.5 \times 10^{-4}\%$	$4 \times 10^{-3}\%$	5.3
	300 secs.	$1.6 \times 10^{-3}\%$	$1.1 \times 10^{-2}\%$	6.9
	200 secs.	$4.4 \times 10^{-3}\%$	$2.9 \times 10^{-2}\%$	6.6

It is evident that the C_x activity of the Koch Light Cellulase is about six times higher than that of Meicelase P.

Naturally, mixtures of various enzymes may be used, as long as they also contain cellulolytic enzymes. Thus for example, amylases, proteases and lipases may be used in conjunction with the cellulolytic enzymes. However, care should be taken that those enzymes are used that do not attack the cellulolytic enzymes and that are active in the same pH range as the cellulolytic enzymes. Cellulolytic enzymes are e.g. very resistant to proteolytic enzymes, and can therefore satisfactorily be used together with these. Most cellulolytic enzymes nowadays available have a pH optimum between 2 and 10. Many types of cellulases derived from fungi have a pH optimum of about 5. Above pH 7 their activity is normally greatly reduced, and therefore the cellulolytic enzymes derived from fungi should be used in the present invention in an acid medium. Bacterial cellulases, such as those derived from Pseudomonas fluorescens have, however a pH optimum of 7—8, and there are also cellulases with a pH optimum of 10, e.g. those derived from a mollusc, Dolabella sp., as described in Biochemical Journal 99, (1966), page 214. Examples of suitable enzymes to be used with fungal and bacterial cellulolytic enzymes are lipases, such as those derived from bacteria, e.g. Candida lipolytica, or from fungi,

e.g. Aspergillus, or from the pancreas; amylases such as those obtained by submerged fermentation of a particular strain of Bac. subtilis (commercially available from Royal Fermentation Industries, Delft, Holland, under the trade name Maxamyl® and from NOVO Industri A/S, Copenhagen, Denmark, under the trade name NOVO-amylase), and proteases, such as papain, trypsin, bacterial proteases of the subtilisin type, e.g. from Bac. subtilis, commercially available e.g., Alcalase® (ex NOVO) and Maxatase® (ex Royal Fermentation Industries), and Rhozyme J—25 (ex Rohm and Haas Cy), a powder mixture of a proteolytic enzyme and a minor amount of diastase, precipitated on corn starch as the carrier material for the enzymes.

The amount of cellulolytic enzymes to be used is of course dependent upon a number of factors, such as, in particular, their activity, duration of the fabric treatment, temperature and so on. For most practical purposes the amount of Meicelase P may lie between 0.01—20% by weight of the fabric-treating composition. The equivalent amount, in respect of C_1 activity, of another cellulolytic enzyme preparation may also be used.

The cellulolytic enzymes may be applied to the fabrics in the form of an aqueous solution, adjusted to the desired pH, or they may be formulated as a detergent active material-containing composition to be added to a wash liquor. Such a composition may be a (pre-) soaking composition, or a main wash composition or a rinse composition. It is preferred to apply fungal cellulolytic enzymes during (pre-) soaking, since the normal main wash compositions have a pH unsuitable for the incorporation of fungal cellulolytic enzymes. Since, furthermore, rinse compositions are normally acid, the cellulolytic enzymes with a pH optimum in the acid range can be used advantageously in such rinse compositions too.

It has been found that optimum results can be obtained with a combination of cellulolytic enzymes in a pre-wash step, and any suitable known rinse conditioning composition in the rinse step after the main wash. Often the results thus obtained are superior to either treatment alone.

The compositions of the invention may be in any suitable physical form, such as liquids, tablets, powders, noodles, and ribbons.

The compositions of the invention may comprise other constituents that are desirable in such softening compositions. Normally, a buffer system should be present if cellulolytic enzymes with an acid pH optimum are used in the compositions. In such a case, the minimum quantity of buffer should be used, in order to avoid as much as possible a drop in pH value of the main wash liquor due to the overflow of the (acid) pre-soak liquor to the alkaline main wash liquor. Too great a drop in pH value would impair the detergency of the main wash liquor. Citric acid, or any other suitable buffer system, proved to be satisfactory. More citric acid, with some sodium citrate, may also be used. Other suitable buffer systems are fumaric or benzoic acid.

The compositions furthermore contain a detergent active material. Although it has been suggested that anionic detergent active materials inhibit cellulolytic enzymes, tests have been carried out to this effect, and some anionic detergent active materials like sodium dodecylbenzenesulfonate proved to be strongly inhibitory to the cellulolytic enzymes. Others, like fatty alcohol sulphates proved to be less inhibitory, and alkyl ether sulphates and -carboxylates proved to give almost no inhibition. The inhibition can be reduced by incorporation of nonionic detergents. Nonionic detergent active materials did not seem to be significantly inhibitory, nor did tertiary amine oxides or sulpho-betaines, and cationic detergent active materials are said in the prior art not to inhibit the cellulolytic enzymes at all. The compositions of the invention therefore preferably contain a nonionic and/or a cationic detergent active material. Suitable examples of anionic, nonionic and cationic detergent active material can be found in Schwartz, Perry and Berch "Synthetic Detergents and Surfactants", Vol. II, 1958. Furthermore, fillers such as sodium sulphate, perfumes, germicides, hydrotropes, opacifiers, solvents, fluorescers, foam-depressors, stabilizing agents for liquids and colouring material may also be present. The cellulolytic enzymes may also be incorporated in normal rinse-conditioning products of which examples are described in Chemistry and Industry, July 1969, page 903.

Example 1.

The following comparative test was carried out. An aqueous buffer solution with pH 5 was prepared from 10.6 g citric acid monohydrate and 18.3 g monosodium dihydrogen orthophosphate $2H_2O$. 5 g pieces of an extremely harsh cotton terry towelling (cloth A) were soaked for a weekend in 200 ml portions of the aqueous solution at 30°C in the presence of additive as given below.

Cloth	Detergent	Enzyme
A	—	—
B	0.1 g secondary C_{11-15} alcohol condensed with 9 moles of ethyleneoxide	—
C	—	0.2 g cellulase (Meicelase P)
D	0.1 g secondary C_{11-15} alcohol condensed with 9 moles of ethyleneoxide	0.2 g cellulase (Meicelase P)

The clothes were rinsed in running tap water and then heated to 80°C for 30 minutes and assessed for softness by feeling them. The results given in the order of decreased softness were D>C>B>A. Cloth D was the softest, and Cloth A the harshest. The above test was repeated with conditions identical to those used for test-piece D, with the exception that the cellulase solution used in this test, D_1, had been preheated to 80°C for 30 minutes to destroy the enzymes. After drying the test-pieces, D felt significantly softer than D_1.

Example 2.

Two 5 g test-pieces of a napkin which had been washed many times in an automatic washing machine were compared in the following way: test-piece C was soaked for a weekend in 200 ml of the buffer solution of Example 1, with the addition of 0.2 g cellulase (Meicelase P), and test-piece C_1 in the buffer solution only. After rinsing in running tap water, and heating to 80°C for 30 minutes to dry, the cellulase-treated test-piece C felt considerably softer than the control test-piece C_1.

Example 3.

Four types of new cotton towels A, B, C and D were washed once with a conventional heavy-duty controlled sudsing detergent composition[2)] in a drum-type washing machine. Thereafter, the towels were cut in two, and half of each towel was washed a further 12 times without the washing product using the main wash cycle and not the pre-wash cycle of the washing machine.

A 5 g test-piece of each type of towel that had been washed 13 times was treated with 200 ml portions of the buffer solution of Example 1 with 0 mg, 50 mg, 100 mg, 150 mg and 200 mg of cellulase (Meicelase P), either at 30° for 66 hours or at 40°C for 18 hours. Thereafter, the test-pieces were rinsed in an aqueous solution of 5 g of the conventional heavy-duty detergent composition[2)] per litre and then rinsed twice in water.

In all cases the 40°C/18 hours treatment gave a softer cloth than the 30°C/66 hours treatment. In both series softness increased with increasing enzyme concentration. The treatment of the test-pieces at 40°C for 18 hours with 200 mg cellulase gave a very slightly softer cloth than that treated with 600 mg cellulase in a subsequent similar test, which in turn was considerably superior to that treated with no enzyme at all.

Example 4.

The softening effect of 200 mg cellulase (Meicelase P) at pH's of 5, 6, 7 and 8 on cotton towel test-pieces was assessed by washing new towels and treating test-pieces of these washed towels with the cellulase solution as in Example 3, at a temperature of 50°C for 18 hours. The test-pieces were dried at 20°C/65% R.H. on a line.

At pH 5 a considerable softening effect was obtained; at pH's of 6, 7 and 8 just detectable softening was obtained. The comparisons were made with test-pieces treated at the same pH's without enzymes.

Example 5.

A 40 g test-piece of a cotton towel M1 which had been washed 13 times was treated in a drum-type washing machine for 1 hour at 50°C in a buffer solution at pH 5 with 2.7 g cellulase (Meicelase P). The machine contained 20 litres of water, the enzyme concentration thus being 0.013%. A control cloth M2 was treated similarly, but without enzymes. After this pre-wash, each cloth was washed with a conventional heavy-duty detergent composition[2)] in a normal wash program followed by the normal rinsing. The clothes were dried on a line at 20°C/65% R.H. The enzyme-treated cloth proved to be much softer than the non-enzyme-treated cloth.

Five cm wide strips of the clothes were cut off and the breaking strain was measured. The results are tabulated below.

	Treatment	Towel No. 1 Breaking strain in Kg		Towel No. 2	
A	1 × washing with normal heavy-duty detergent composition[2])	35.6 35.2 37.8	30.8	34.4 35.8 33.2	34.5
B	1 × washing with normal heavy-duty detergent composition[2]) + 12 further washes in water (cloth B)	33.3		18[x]	
C	Cloth B again washed and treated with cellulase in pre-wash cycle of washing machine	32.9 32.4 29.4	31.8	32.4 32.0 31.4	31.9
C[1]	Cloth B again washed but without cellulase in the prewash cycle of washing machine.	33.8 30.3 33.3	32.5	34.8 35.4 26.8	31.8

[x]probably due to unknown malfunctioning of machine during test.

These tests indicated that the loss in strength one would expect accompanying the cellulase treatment was either small or zero. In an additional test, 55 g cotton (13 times washed) was treated for 1 hour at 50°C in the washing machine with 2.7 g cellulase, buffered at pH 5. No reduction in the degree of polymerization of cotton could be found.

Example 6.

Example 4 was repeated (temperature 50°C, soaking time 18 hours: pH 5, drying at 20°C/65% R.H.) with 0.08 g cellulase ex Merck (activity 20 mU/mg), 3.4 g cellulase C 7377 ex Sigma or 3.4 g cellulase 36 concentrated ex Rohm and Haas. The same softening as with 0.2 g Meicelase P was obtained.

Example 7.

The test articles were four cotton towels that had been harshened by 14 washes in a drum-type washing machine. Two of the towels were treated at 20°C with a conventional rinse conditioner[1)] in aqueous solution for 10 minutes. The liquor to cloth ratio was 7:1 and the cloth to rinse conditioner ratio was 70:1. After line drying these articles (A) were substantially softer than the two articles (B) which had not been treated with the conventional rinse conditioner.

The test proceeded as follows in the drum-type washing machine:

	Test articles	Pre-wash	Main wash	Rinse
A	Towels treated with conventional rinse conditioner (fairly soft)	Buffer	conventional heavy-duty detergent composition	Water
B	Towels not treated with conventional rinse conditioner (harsh)	Buffer + Meicelase P	„	Water

The conditions in this test were as follows:

Pre-wash

Weight of articles	325 g
Buffer (pH=5)	51.5 g citric acid monohydrate + 91.5 g $Na_2HPO_4.2H_2O$
Meicelase P used (test B)	20 g
Duration/Temperature	15 min heating from cold to 50°C. then 45 min at 50°C.

Main wash

Normal programme	130 g conventional heavy-duty detergent composition[2])

Rinse

Normal programme.

At the end of the test the two articles B were assessed by three independent observers as being softer than the two articles A.

This experiment demonstrated that the softening effect obtained with the cellulolytic enzymes is more persistent than that obtained with a conventional rinse conditioning product.

[1])The conventional rinse conditioning (=fabric softening) product contained the following constituents:

	%
di-tallow dimethylammonium chloride	2.5
a cationic fatty acid amide derivative, known under the trade name Ceranine HC	2.5
citric acid	0.25
colouring material, perfume, water	q.s.

[2])The conventional heavy-duty detergent composition was:

	%
sodium dodecylbenzenesulphonate	4.5
nonylphenol condensed with 14 moles of ethylene oxide	3
coconut fatty acid diethanolamine	1.2
sodium stearate	3.3
sodium triphosphate	32
sodium silicate	3.7
sodium sulphate	9.1
sodium carboxymethylcellulose	1.0
sodium perborate	30
fluorescer, sequestering agent, perfume, moisture, etc.	q.s.

Example 8.

The test articles were cotton towels that had been harshened by 14 washes in the drum-type washing machine. Four pairs of articles were treated in the washing machine with products as indicated below:

Article	Meicelase P	Conventional rinse conditioner 1)
A	—	—
B	—	42 g
C	20 g	42 g
D	20 g	—

The conditions were as follows:

weight of test articles	325 g
weight of ballast articles	3 kg
buffer solution (pH = 5)	51.5 g citric acid monohydrate + 91.5 g $Na_2HPO_4.2H_2O$
duration/temperature	15 min to 50°C. then 45 min at 50°C.

At the end of the treatment the articles were removed from the machine and dried on a line. The four pairs of articles were ranked by three observers in order of increasing softness, with 1 being assigned to the harshest cloth and 4 being assigned to the softest cloth in each of the two series.

Results:

Cloth	Rank totals
A	6
B	9
C	22
D	19

It was concluded that the enzyme was effective for softening and that the combination enzyme/conventional rinse conditioner was apparently superior to either alone.

Example 9.

Cotton towels harshened by 10 washes in a drum-type washing machine were used.

These articles were then treated in the washing machine with the following products:

Article	Pre-wash	Main wash	Rinse
I	Buffer, pH 5 (citrate/phosphate) 15 g conventional rinse product1) 2 g Meicelase P volume of solution 18l	130 g conventional heavy-duty detergent composition2)	(a) — (b) 42 g conventional rise product 1)
II	Buffer, pH 5 (citrate phosphate) 15 g conventional rinse product 1) volume of solution 18l	130 g conventional heavy-duty detergent composition 2)	(a) — (b) 42 g conventional rinse product 1)
III	Buffer (pH 5 using citrate/phosphate) volume of solution 18l	130 g conventional heavy-duty detergent composition 2)	42 g conventional rinse product 1)

For articles I (a), II (a) and III the normal pre-wash, main wash and rinse cycles of the washing machine were used, with 3 kg ballast.

Articles I (b) and II (b) were obtained by taking pieces of articles I (a) and II (a) and treating these pieces with the conventional rinse conditioner and ballast goods in an additional rinse in the washing machine.

All articles were line-dried and felt to assess softness. The results were:

(i) II (b) and III were of similar softness, indicating that the use of the conventional rinse conditioner in the pre-wash had no big influence on the final softness at the end of the washing process.

(ii) I (a) was softer than II (a), showing that the cellulase in the pre-wash has a softening effect which does last through the main wash. Cloth I (a) was, however. not as soft as cloth III.

(iii) II (b) was softer than II (a) and I (b) was softer than I (a). Thus, as expected, the conventional rinse conditioner softened in the rinse and moreover softened an article which had previously been treated with cellulase.

(iv) I (b) was substantially softer than II (b), indicating that the cellulase treatment in the pre-wash and the conventional rinse conditioning treatment in the rinse of cloth I (b) were both contributing to the softness of this cloth.

Example 10.

Buffer solutions containing 2.04 g citric acid and 3.66 g $Na_2HPO_4.2H_2O$ in 200 ml water were prepared. 200 mg cellulase (Meicelase P) was dissolved in this solution. Four pieces of Krefeld Standard cotton test pieces (total 10.5 g), about 6 cm wide, and 25 cm long, were shaken with the buffer solutions at 40°C automatically. Each day the cloth samples were placed in fresh buffered Meicelase P solutions, as it had been found that after 24 hours under these conditions only 40% of the original enzyme activity remained. The incubation times were 0, 1, 3 and 7 days, and the cloths (in quadruplicate) were tested for strength with the W.P.M. Type ZT 100 machine from V.E.B. Thüringer Industriewerke. The stretching speed was 9mm/second, and the distance between the clamps was 10 cm. There was a linear correlation between loss in strength and time, and the rate of loss of strength was 0.6% per hour.

Example 12.

It has been demonstrated that better softening is obtained with higher C_1 concentration, if the C_x is kept constant.

At a liquor/cloth ratio of 40:1, pieces of harsh cotton were soaked in buffer solutions at pH 5 overnight. The initial temperature was 50°C. After soaking, the articles were rinsed with an aqueous solution containing 5 g/l of a conventional heavy-duty detergent[2)] and then thoroughly with tap water. They were finally dried at 20°/65% RH.

Results

Enzyme (% by weight of buffer solution	Ranking order for softness (1 = softest; 4 = harshest)	Relative C^x content
a) 0.1 % Meicelase P	1	5
b) 0.02% ,,	2	1
c) 0.1% Koch Light enzyme	3	30
d) 0.015% ,, ,, ,,	4	4.5

a) and d) contained practically the same amount of C_x, and for comparison purposes b) and c) were also included in this test series, in which c) contained abt. 30 times as much C_x as b). b) still ranked softer than c).

Example 12.

New cotton towels were washed once in a (4 kg) drum washing machine using 100 g of the conventional heavy-duty detergent composition[2)] in the normal pre-wash cycle and 100 g in the normal main wash cycle.

Half of these towels were dried and reserved (cloths W, each weighing 130 g). The other half were washed a further 25 times in the washing machine without detergent using the normal main wash cycles. These cloths were coded G, and each weighed 130 g.

Washing tests

The W article and one G article were placed together with 3 kg of normally soiled wash goods in the drum of the machine which was allowed to proceed through the normal pre-wash and main wash and rinse cycle. Seven such tests were done in parallel with seven different combinations of pre-wash product,

main wash product, and rinse product, as indicated below. When a rinse product was used it was added at the beginning of the last rinse. At the end of each washing cycle the towels W and G were dried on a line. 14 repeat cycles were carried out.

Pre-wash	Main wash	Rinse	softness ratings W	softness ratings G
conventional heavy-duty detergent composition 2)	conventional heavy-duty detergent composition 2)	—	0	−2
conventional heavy-duty detergent composition 2)	conventional heavy-duty detergent composition 2)	conventional rinse conditioner 1) x	3	3
buffer (pH of 5) volume of solution 221 containing 51.5 g citric acid 91.6g $NaH_2PO_42H_2O$	conventional heavy-duty detergent composition 2)	—	2	2
buffer + 2 g enzyme (as above) (as above)	conventional heavy-duty detergent composition 2)	—	5	5
buffer + 20 g enzyme (as above (as above)	conventional heavy-duty detergent composition 2)	—	8	6
buffer + 2 g enzyme (as above) (as above)	conventional heavy-duty detergent composition 2)	conventional rinse conditioner 1) x	10	7

x only 1/1.7 of the recommended concentration was used (i.e. 25 g in the rinse)

rating: 1 = feel of the 25 times washed cloth (harsh) 10 = feel of the once washed cloth (soft)

Example 13.

In a drum-type washing machine, using its normal programme, 3 kg dirty wash goods and harsh cotton towels which had been washed 14 times were laundered.

In experiment A, a pre-wash product was used of the following composition:

80 g sodium sulphate
20 g Meicelase P
10 g sec. C_{11-15} alcohol condensed with 9 moles of ethylene oxide
20 g benzoic acid.

The pH at the end of pre-wash was 4.9. The solution volume was 22 l. In the main wash 180 g of the conventional heavy-duty detergent composition 2) was used. The pH at the end of the main wash was 9.4. The wash goods and towel were rinsed with tap water and dried on a line. In experiment B, the pre-wash was carried out with 60 g of the conventional heavy-duty detergent composition 2) (pH at the end of the pre-wash was 9.4) and the main wash with 150 g of the same product (end pH 9.6).

The rinse was carried out in the normal rinse cycle with tap water and the wash good and towel were dried on a line.

The towel in Experiment A felt noticeably softer after washing than in Experiment B.

WHAT WE CLAIM IS:—

1. Method for reducing the rate at which cotton-containing fabrics become harsh or reducing the harshness of such fabrics comprising applying cellulolytic enzymes as herein before defined to the fabrics.

2. Method according to Claim 1, wherein the cellulolytic enzymes are applied in a soaking process.

3. Method according to Claim 1, wherein the cellulolytic enzymes are applied in a rinse process.

4. Method according to Claims 1—3, wherein fungal cellulolytic enzymes are applied in an aqueous liquor with an acid pH value.

5. Method according to Claim 4, wherein the pH value of the aqueous liquors is 5.

6. Method according to Claim 1, wherein the fabrics are treated with cellulolytic enzymes in a pre-soaking step, washed, and subsequently rinsed with a rinse conditioner.

7. A method as claimed in Claim 1, substantially as herein described.

8. A composition for use in a method according to Claims 1—7, said composition comprising a cellulolytic enzyme as hereinbefore defined, and a detergent active material.

9. A composition according to Claim 8, wherein the detergent active material is a cationic detergent active material.

10. A composition according to Claim 8, wherein the detergent active material is a nonionic detergent active material.

11. A composition according to Claims 8—10, wherein the cellulolytic enzyme comprises a fungal cellulolytic enzyme, said composition having an acid pH value.

12. A composition according to Claim 11, wherein the composition has been buffered at an acid pH value by means of citric acid.

13. A composition according to Claim 12, wherein the composition has been buffered at an acid pH value by means of fumaric or benzoic acid.

14. A composition as claimed in Claim 8, substantially as described in any of Examples 1D and 13A.

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